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PATENT

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INVENTION DISCLOSURE

PAGE ONE OF

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Descriptive Title of Invention:

A Method for Etching High Aspect Ratio Features in III-V-based Semiconductors for Optical and Electrical Devices

Name of Project: Photonic Crystals and III-V Integration

Product Name or Number:

Was a description of the invention published, or are you planning to publish? If so, the date(s) and publication(s):
I would like to publish this by the end of 2003

Was a product including the invention announced, offered for sale, sold, or is such activity proposed? If so, the date(s) and location(s):
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Was the invention disclosed to anyone outside of AGILENT TECHNOLOGIES, or will such disclosure occur? If so, the date(s) and name(s):
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If any of the above situations will occur within 3 months, call your IP attorney or the Legal Department now at 1-635-6861 or 970-635-6861.

Was the invention described in a lab book or other record? If so, please identify (lab book #, etc.)

Run sheets on Etch system, email and report Dater

Was the invention built or tested? If so, the date: YES

Was this invention made under a government contract? If so, the agency and contract number:
NO

Description of Invention: Please preserve all records of the invention and attach additional pages for the following. Each additional page should be signed and dated by the inventor(s) and witness(es).

- A. Prior solutions and their disadvantages (if available, attach copies of product literature, technical articles, patents, etc.).
- B. Problems solved by the invention.
- C. Advantages of the invention over what has been done before.
- D. Description of the construction and operation of the invention (include appropriate schematic, block, & timing diagrams; drawings; samples; graphs; flowcharts; computer listings; test results; etc.)

Signature of Inventor(s): I (we) hereby submit this disclosure on this date:

00462062

Laura W. Mirkarimi

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Agilent Labs - CORL

Employee No.

Name

Signature

Telnet

Mailstop

Entity & Lab Name

Employee No.

Name

Signature

Telnet

Mailstop

Entity & Lab Name

Employee No.

Name

Signature

Telnet

Mailstop

Entity & Lab Name

Employee No.

Name

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Signature of Witness(es): (Please try to obtain the signature of the person(s) to whom invention was first disclosed.)

The invention was first explained to, and understood by, me (us) on this date ____

Full Name

Annette Grot

Signature
Annette Grot

Date of Signature

Full Name

Scott William Corzine

Signature
Scott William Corzine

Date of Signature

Inventor & Home Address Information: (If more than four inventors, include add. information on a copy of this form & attach to this document)

Inventor's Full Name

Laura Wills Mirkarimi

Street

2155 Kilcare Road

City

Sunol

State

Zip

CA

94586

State

Zip

Do you have a Residential P.O. Address? P.O. BOX

City

Citizenship

Greeted as (nickname, middle name, etc.)

Inventor's Full Name

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City

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A Method for Etching High Aspect Ratio Features in III-V-based Semiconductors for Optical and Electrical Devices

Laura W. Mirkarimi

A. Prior Art and Solutions:

There is significant interest in the ability to etch high aspect ratio features with a large $>88^\circ$ sidewall in InP based materials for optical devices. The general trend in etching high aspect ratio features in III-V based Semiconductors both GaAs and InP is to use dry etching and implement a high energy density plasmas such as Inductively Coupled Plasma (ICP) or Electron Cyclotron Resonance (ECR) or Chemically Assisted Ion Beam Etching. All of these techniques provide a combination of physical and chemical etching. To date, most work has focused on using Cl chemistry based etches.

I can not find any specific references that seem to overlap with my invention. Here are the citations that I did find in my searches that provide a view of prior art and etch process patents [1-2]. I have included numerous journal articles for reference that discuss the prior art chemistry and approaches using Cl, Ar, CH₄, H₂, SiCl₄, BCl₃ and etc [3-11]. Additional patents on wet etch HBr solutions were found; however, this is a completely different application and invention [12,13].

B. Problems Solved by the Invention:

This invention takes a radical departure from the prior solutions and implements a standard Reactive Ion Etching technique with novel chemistry to achieve these goals. The use of RIE mode etching enables the amplification of selective etching between the mask and material of etched. One of the main difficulties in attempting to use the state of the art etch solutions to fabricate photonic crystals is that the mask material would degrade well before the desired etch depth was achieved. The submicron feature size is the critical parameter that requires a new etch solution for fabricating high aspect ratio etches $> 5:1$. The small feature size and geometry of the photonic crystal lattice provides many thin walled

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features in the mask that can be attached by the ions in the plasma and physically sputter the mask away. As the mask erosion proceeds, the feature of interest in the substrate becomes deformed. If the mask erosion is severe, it is possible that the desired etch depth can not be obtained before the entire mask erodes.

Choosing the appropriate chemicals is one of the most important issues regardless of what type of etch system is used. The novel chemistry that we used is a bromine based chemistry rather than an Cl based chemistry to etch the InP. The combination of the CH₄, H₂ and HBr enables a high chemical selectivity between the mask and the etched material. Additionally, the HBr provides a passivation on the vertical surfaces which enables the high aspect ratio etch result of > 20:1.

C. Advantages of the invention

Replacing the ever-popular chlorine chemistry with bromine has the two main advantages that the In_xBr_y and Ga_xBr_y products are more volatile than the In_xCl_y and Ga_xCl_y and the HBr appears to have a self-passivation on the vertical surfaces which enables one to achieve very vertical surfaces. Therefore, high aspect ratio >10:1 features can be fabricated in InP to construct numerous electronic and optical devices. The sidewall etch is quite smooth and extremely vertical, both of which are important for photonic crystal lattices. This invention is widely applicable to the III-V community. The regions of high etch rates are defined for alternative etch chemistries to make a variety of optoelectronic devices which require vertical side walls and substantial etch depths such as microdisc resonators, VCSELs, lasers, waveguides as well as photonic crystals and others. It may enable the work that is currently done in a high density plasma systems to be done in standard RIE systems simply by choosing the right chemistry. Note that every university or lab typically has an RIE system; however, they may not have the more exotic high density plasma systems such as ICP or ECR systems. The novel chemistry chosen in this work will enable

many new optical and electronic devices to be fabricated with improved performance.

D. Description of the Invention

A thin SiO₂ or Si₃N₄ or other appropriate mask material is grown onto a III-V substrate or III-V epitaxial layers. A III-V material is one that consists of combinations of Group III elements such as Al, Ga, In, B and group V elements being N, P, As. The mask choice is an important part of the invention because of the selectivity that this invention offers with the SiO₂ and Si₃N₄ masks. The mask is then defined by a lithographic technique such as e-beam or other appropriate lithography for sub-micron features Figure 1(a). The lithographic pattern is transferred into the oxide with a dry etch technique using prior art techniques such as CHF₃ in a reactive ion etch system Figure 1(b). The sample is then placed in a Reactive Ion Etch system. Chemistries consisting of CH₃, H₂ and HBr are necessary to transfer the defined feature into the semiconductor. These three constituents are the primary gases required to obtain the desired high aspect ratio etching. I have attached a report that discusses some of the parameter space which has been investigated to date. That report shows how the etch rates vary with the concentration of HBr and Cl in the submicron feature area and compares this to the large micron sized features (identified as the field etch). That report describes in detail the conditions that are used to obtain these results. Below is the range of conditions that this invention should include:

DC Bias = 100-500V

P=50-200 Watts

Temp ≥ 60

HBr=2-75%

CH₄=0-25%

H₂ = 0-12.5%

Pressure= 1-30mTorr

The important ratios that have been determined thus far are $\text{CH}_4:\text{H}_2 = 1:1$ to $2:1$ and the $\text{HBr}:\text{CH}_4$ may range from $1:4$ to $4:1$. The attached report dated contains many details that can be incorporated in the claims of this invention.

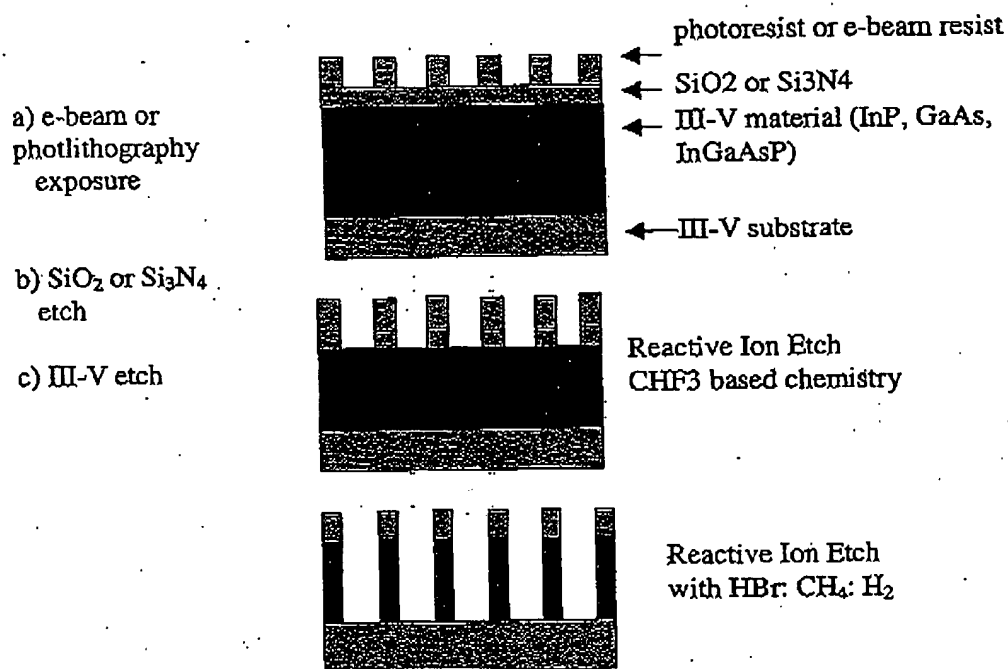


Figure 1: Schematic of Process Flow for the Etching

References

- [1] USP 5,338,394 Method for etching Indium based III-V compound Semiconductors (attached)
- [2] USP 5,624,529 Dry Etching Method for Compound Semiconductors (attached)
- [3] H-Y.Chen and H. Ruda, J. Vac. Sci. Tech. B 19(5), p.1694, (Sept 2001).
- [4] K.Y. Hur, et al, J. Vac. Sci. Technol. B 12(3), p.1410 (May 1994).
- [5] Y. Feurprier et al J. Vac. Sci. Technol. B 15(5), p. 1733 (sept 1997).
- [6] A. Matsutani et al., Jpn. J. Appl. Phys 41, pp. 3147-3148. (2002).
- [7] A. Masutani et al., Jpn. J. Appl. Phys. Vol. 40, pp. 1528 (2001).
- [8] A. Matsutani et al. Jpn. M. Appl. Phys. Vol. 38, pp 4260 (1999).
- [9] K.K. Ko and S.W. Pang, J. Vac. Sci. Technol. B 11(6) 2275 (1993).
- [10] J. E. Parmeter et al, J. Vac. Sci. Technol. B 14(6) 3563 (1996).
- [11] D.L. Melville et al., J. Vac. Sci. Technol. B 11(6), pp2038 (1993).
- [12] USP 5,723,360 Method of processing an epitaxial wafer of InP or the like
- [13] USP 5,304,283 Proces for producing buried stripe semiconductor laser using....

To: Mike Tan
Re: Etch Data on InP

From: Laura Mirkarimi
Date:

I have made some progress in developing a selective etch for InP photonic crystals using a SiO₂ hard mask. While I am sure that there are still improvements to be made in terms of the sidewall roughness reduction and wall angle, the basic chemistry and trends are encouraging. My criteria differs from previous work in InP in our lab, the feature sizes of my structures are typically 260 nm with pitches of 400nm and etch depths as large as 2µm which is an aspect ratio of > 10:1 at the wall height to wall thickness.

Initial etch experiments in InP focused on using the ICP etcher with Cl₂, CH₄ and H₂ chemistries. While I did optimize a process with this chemistry, the results were disappointing. The main problem was the competition between mask erosion while etching to the appropriate depth in the InP. Figure 1 shows the results that I obtained with this approach.

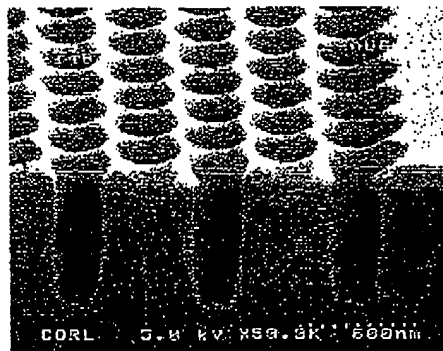


Figure 1: Cl₂/CH₄/H₂ = 6/8/4.5 (sccm); RF1/RF2= 150/998 Watts;
3 mTorr; ; SiO₂= 300nm

I developed an etch process for Si photonic bandgap waveguides using HBr, which has a very high selectivity between SiO₂ and Si. Additionally, HBr appears to passivate the sidewalls as you etch. I had high hopes for HBr as an etchant for the InP photonic crystals, which require much larger etch depths than our Si photonic crystal waveguides. Thanks to David Lin for adding the HBr gas panel functionality, I was able to conduct the following tests.

The first experiments were a slight variation from typical InP etch conditions in the ICP. I did perform some experiments with Cl₂/HBr/CH₄/H₂; however, these results were similar to the experiments using Cl₂/CH₄/H₂ which were shown in Figure 1. Next I removed the Cl₂ and used HBr/CH₄/H₂ with the ICP mode and found similar results to that of the Cl₂/CH₄/H₂. The scanning electron micrograph in Figure 2 shows the cross

section of a photonic crystal etched with the new chemistry in the ICP mode. Again, this etch condition attacks the SiO₂ hardmask, thereby limiting the etch depth in the InP.

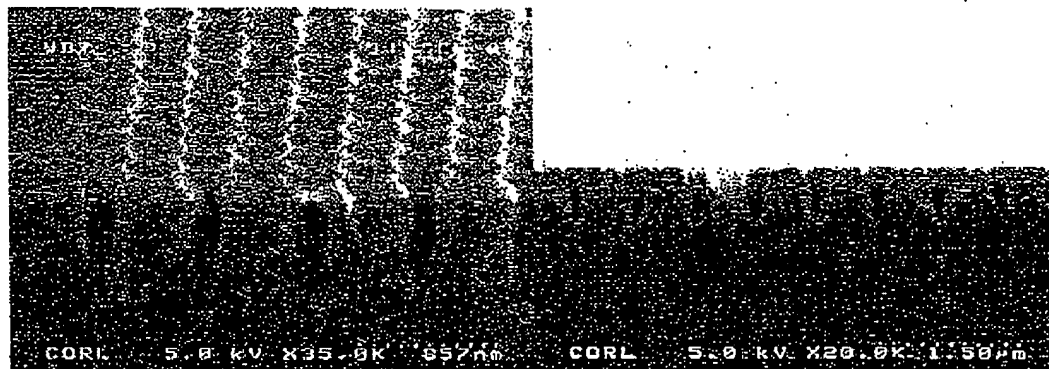


Figure 2: a) SiO₂ mask remains; however it is thinning and the sidewalls are not verticle. RF1 = (150); RF2= 800; Temp set = 60; HBr/CH₄/H₂=6/8/4.5; 4mTorr; time=2.5'; b) SiO₂ mask has been etched away (5 minute) RF1 = 150 Watts; RF2= 500 Watts; Temp= 60; HBr/CH₄/H₂=6 sccm/8 sccm/ 4.5 sccm; 4mTorr; time= 5'.

I abandoned the above approach and began evaluating the HBR chemistry using the RIE mode in the ICP etch system. With this approach, I obtained some encouraging results. Figure 3 shows the cross sectional view of a photonic crystal waveguide etched with the following conditions: DC Bias = 458; P=180 Watts; Temp = 60; HBr/CH₄/H₂=8/8/4.5

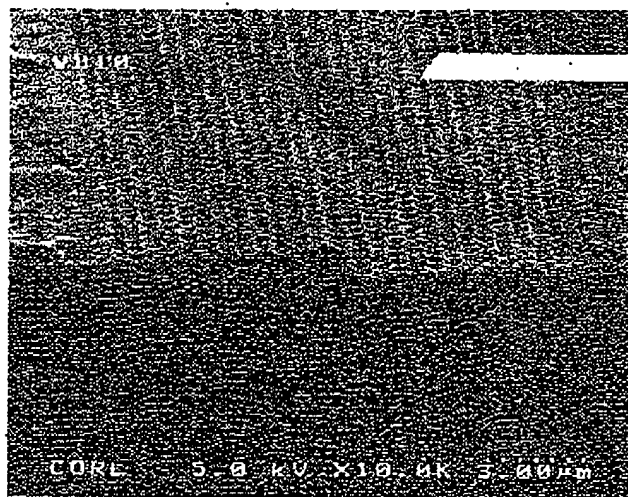


Figure 3: Cross sectional view of SiO₂/InP. Vertical side walls and mask retention look good.

Recently, I have evaluated the influence of the pressure and percent CH₄ on the etch in the hope to fine tune the process to obtain deep etches in InP with smooth sidewalls and little polymer deposition. During these experiments I measured the etch rate of InP in the field (a 300um square) by a surface profilometer and estimated the etch rate in the photonic crystal (PC) area by cross sectional scanning electron microscopy. The etch rate of InP in the field is much higher than that in the PC region, which is likely due to diffusion issues with the high aspect ratios (Figure 4). The maximum etch rate in both the field and the PC region is achieved at 4 mTorr.

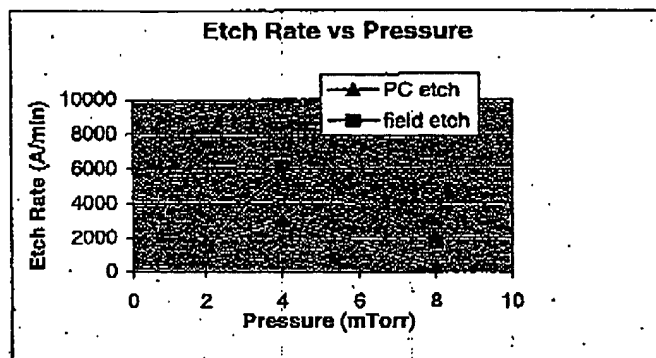


Figure 4: Graph of the Etch rate in the photonic crystal region and the field are evaluated as a function of pressure in the system

CH₄ plays an important role in the etch process. When no CH₄ is used, the HBR removes the SiO₂ mask for very long etches. Additionally, too much CH₄ causes a significant polymer build-up in the field and can eventually lead to closing over the small holes in the photonic crystal (Figure 5)

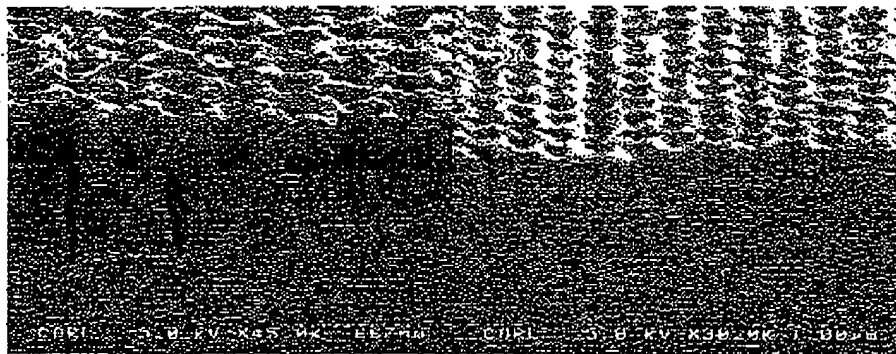


Figure 5: 40% CH₄ in the gas mixture. CH₄/H₂/HBr= 8/4.5/8; 2mTorr ; RF1=150 Watts.

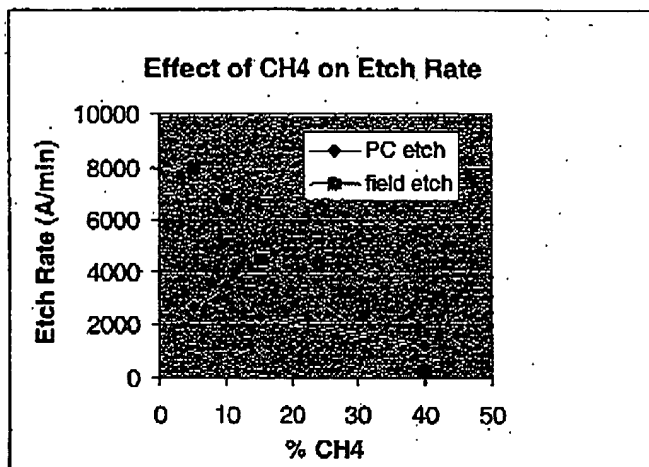


Figure 6: Etch rate of InP is shown as a function of the % CH4. As the %CH4 is increasing the %HBr is decreasing.

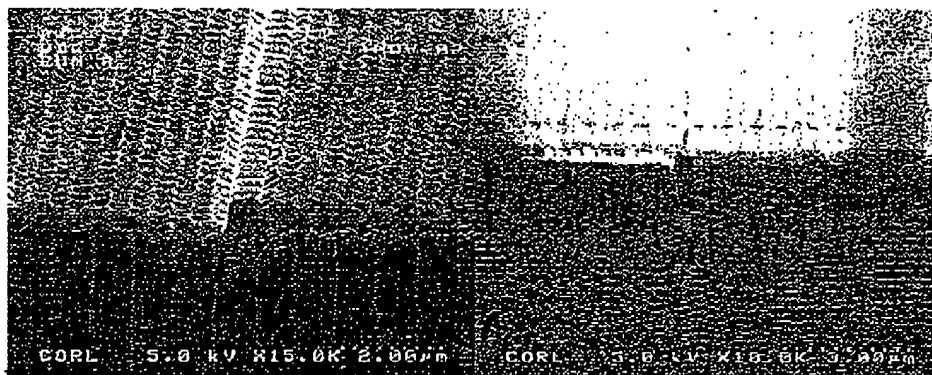


Figure 7: The oxide mask remains in tact and vertical sidewalls were obtained.

In conclusion, I have developed a selective etch for InP using HBr, CH4 and H2 using a standard RIE mode in the IPC etcher. The selectivity between the InP and SiO2 is typically greater than 40:1. This approach is quite promising for fabricating nanoscale InP based structures with high aspect ratios.

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